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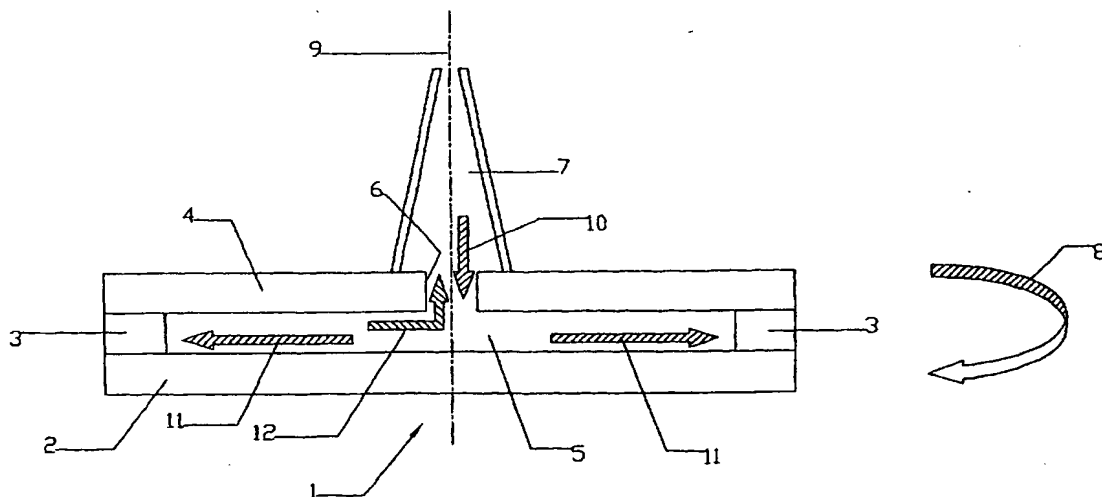
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(54) Title: **METHOD AND APPARATUS FOR DYING A LAYER OF NANOCRYSTALLINE MATERIAL**



(57) Abstract: Method for dyeing a layer of a nanocrystalline material on a substrate using a liquid dye, comprising the successive steps of (i) providing said layer on a substrate, (ii) providing an apparatus for dyeing said layer, which apparatus comprises at least a supply container for the liquid dye, a closable substrate holder provided with at least one inlet and at least one outlet for a substrate provided with a layer of nanocrystalline material, and conduit and circulation means for causing the liquid dye to circulate through the supply container and the substrate holder, (iii) placing the substrate with said layer in the substrate holder and closing the substrate holder, and providing a liquid dye in the supply container, and (iv) causing the liquid dye from the supply container to circulate for a determined time through the substrate holder, and apparatus for performing this method.

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METHODE AND APPARATUS FOR FILLING A DYE SENSITIZED SOLAR CELL WITH ELECTROLYTE

The invention relates to a method for filling with liquid a semi-manufactured product for a liquid-containing photovoltaic element, which semi-manufactured product comprises at least one plate-like work electrode and a plate-
5 like counter-electrode adhered thereto by means of a vapour and liquid-tight peripheral edge, wherein between the work electrode, the counter-electrode and the peripheral edge a space is provided for receiving a liquid.

A liquid-containing photovoltaic element is known from
10 the American patent no. 5,350,644. The known photovoltaic element comprises a work electrode which is formed by a layered structure of at least a first electrically conductive layer, which is for instance deposited on a first substrate, or even forms a first substrate, a layer of crystalline metal
15 oxide semiconductor material deposited on the first electrically conductive layer, a counter-electrode which is formed by a transparent second electrically conductive layer deposited on a transparent second substrate, and an electrolytic liquid held between the layer of semiconductor
20 material and the second electrically conductive layer. In practical situations use is usually made of a glass plate for the first and second substrate.

During manufacture of a liquid-containing photovoltaic element it has to be filled with the electrolytic liquid,
25 which has slightly viscous properties. The filling takes place after the first and second substrate with the respective layers deposited thereon are adhered to each other by means of a vapour and liquid-tight peripheral edge of a thermoplastic adhesive material, wherein between the two
30 substrates and the peripheral edge a space is created for receiving the liquid.

The filling takes place according to the prior art by arranging two holes in one of the substrates, injecting

electrolytic liquid into the space through a first hole and allowing air to escape through the second hole, until the space is completely filled, and subsequently sealing both holes. The arranging of two holes in one of the substrates is
5 mechanically undesirable, and moreover has an adverse effect on the cost-price of the photovoltaic element.

According to an alternative known method, a hole is arranged in one of the substrates, whereafter the element to be filled is placed, with the substrate provided with the
10 hole directed upward, in a vacuum chamber to be vacuum-pumped. A holder open on its underside, to be filled with electrolytic liquid, is then placed on the substrate around the hole, the vacuum chamber is vacuum-pumped wherein the space between the substrates is likewise vacuum-pumped, and
15 air is finally admitted into the vacuum chamber, wherein electrolytic liquid is pressed out of the holder via the hole into the space between the substrates. This method is time-consuming and, due to the inherent use of an expensive vacuum pump which is exposed to oxidizing constituents in the
20 electrolytic liquid, has an adverse effect on the cost-price of the photovoltaic element.

It is an object of the invention to provide a method for filling a semi-manufactured product for a photovoltaic element with an electrolytic liquid, according to which it is
25 possible to suffice with arranging a hole in one of the substrates of the semi-manufactured product for admitting the liquid into the space between the substrates, without herein having to vacuum-pump this space.

It is a further object to provide such a method which
30 enables the manufacture of a semi-manufactured product in rapid and cost-saving manner.

These objectives are achieved with a method of the type stated in the preamble which according to the invention comprise the steps of (iii) placing the semi-manufactured
35 product with the filling opening directed upward on a horizontally placed rotatable disc in a manner such that the filling opening extends round the rotation axis of the disc

within a circle of a predetermined radius R , (iv) placing around the filling opening on the semi-manufactured product a holder with a flat underside and provided with an opening, wherein the opening has at least an internal diameter $2R$ and
5 the outer diameter of the holder on its underside is smaller than the smallest surface area dimension of the semi-manufactured product, (v) introducing liquid into the holder and causing rotation of the disc with the semi-manufactured product and the holder placed thereon at a rotation speed and
10 for a period such that the space in the semi-manufactured product is completely filled with liquid under the influence of centrifugal forces and the gravitational force acting on the liquid, and (vi) sealing the filling opening.

In order to enhance a good mechanical contact between the
15 underside of the holder for placing in step (iv) and the semi-manufactured product, according to an embodiment of the method the holder is placed under pre-pressure on the semi-manufactured product.

In an advantageous embodiment a liquid-tight seal is
20 provided between the underside of the holder and the semi-manufactured product during placing of the holder in step (iv), so that no liquid can leak out of the holder under the influence of a centrifugal force between the underside of the holder and the semi-manufactured product.

25 The rotation speed in step (v) of a method according to the invention amounts to for instance at least 2000 rpm (revolutions per minute), and preferably amounts to at least 4000 rpm. At this latter rotation speed, filling of the semi-manufactured product is on the one hand completed in a
30 sufficiently short period of time, while on the other the centrifugal forces occurring on the constituent parts of the semi-manufactured product are not so great that these forces can result in damage to the semi-manufactured product.

In another embodiment of a method according to the
35 invention, a pulsating underpressure is applied in the holder during introducing of the liquid into the holder during step (v). It has been found that by applying a pulsating

underpressure in the holder the flow rate of the liquid through the filling opening is increased compared to the flow rate in the absence of this pulsating underpressure, under otherwise identical conditions. In an advantageous embodiment 5 the underpressure to be applied amounts to about 10 kPa.

The method according to the invention is particularly suitable for filling a semi-manufactured product which comprises at least one glass substrate.

The invention further relates to an apparatus for 10 performing the above-described invented method, which apparatus comprises a rotatable disc which in the operating situation is placed horizontally for placing thereon of a semi-manufactured product with a filling opening directed upward, and a rotatable liquid holder which can be placed 15 around the filling opening on the semi-manufactured product and which is flat on its underside and provided with an opening, wherein the outer diameter of the holder on its underside is smaller than the smallest surface dimension of the semi-manufactured product.

20 In one embodiment the holder is symmetrical about an axis of symmetry and the holder can be placed in a manner such that the axis of symmetry coincides with the axis of rotation of the disc.

A symmetrical holder has the advantage that it is in 25 balance during the rotation and does not cause any vibrations in the underlying semi-manufactured product.

The holder has for example 2-fold, 4-fold or 6-fold symmetry, i.e. when rotated through an angle of respectively $2\pi/2$, $2\pi/4$ or $2\pi/6$ the holder is carried over into itself.

30 The holder is preferably rotation-symmetrical.

In a holder of an embodiment of an apparatus according to the invention, the interior of the holder has in a longitudinal section a downward widening conical progression. Such a conical progression provides the advantage that pushing of 35 the liquid upward along the inner wall in the holder is counteracted as a result of centrifugal forces.

In another embodiment of an apparatus according to the

invention, the holder is provided with spring means for placing the holder under pre-pressure on the semi-manufactured product, thereby enhancing a good mechanical contact between the underside of the holder and the semi-manufactured product.

In an advantageous embodiment the holder is provided on its underside with sealing means for providing a liquid-tight seal between the underside of the holder and a semi-manufactured product.

10 The invention will be elucidated hereinbelow on the basis of an embodiment of an apparatus, with reference to the drawings.

In the drawings

Fig. 1 shows a schematic vertical section of a semi-manufactured product for a liquid-containing solar cell on which a cone-shaped holder is placed, and

Fig. 2 shows a simplified vertical section of an embodiment of a component with a rotatable liquid holder of an apparatus for filling a semi-manufactured product for a solar cell.

Corresponding parts are designated in the drawings with the same reference numerals.

Fig. 1 shows a semi-manufactured product 1 for a liquid-containing solar cell, with a plate-like work electrode 2, and a plate-like counter-electrode 4 adhered thereto by means of a vapour and liquid-tight peripheral edge 3, wherein work electrode 2, counter-electrode 4 and peripheral edge 3 enclose a space 5 for receiving a liquid therein, and a filling opening 6 is arranged in counter-electrode 4. On counter-electrode 4 there is placed around filling opening 6 a downward widening, conical holder 7 open on its underside, wherein the dimensions are chosen such that the outer diameter of holder 7 does not extend outside the surface area of work electrode 4, and filling opening 6 falls wholly within the opening of holder 7. When the space 5 is filled according to the invention with an electrolytic liquid, the semi-manufactured product 1 and holder 7 are simultaneously

rotated as according to arrow 8 about a vertical rotation axis 9, wherein the liquid is admitted into holder 7 and semi-manufactured product 1, wherein under the influence of the centrifugal force in radial direction as according to
5 arrows 11 the liquid is driven into space 5 while simultaneously displacing air which is present, which escapes through filling opening 6 (as shown by arrow 12).

Fig. 2 shows a filler head 13 of an apparatus for filling a semi-manufactured product for a solar cell, which filler
10 head 13 is assembled from a static and a rotatable part. The rotatable part comprises inter alia a rotation-symmetrical, conical liquid holder 7 widening downward internally, with foot 14, an insert 15 suspended in an insert holder 16 and provided with a continuous axial hole 17 which debouches into
15 liquid holder 7. The static part comprises inter alia a housing, assembled from parts 18, 19 which are fixed to each other with a bolt 25, closed by an upper cap 20 which is fixed with a bolt 32 to housing 19 and which is provided with a liquid inlet opening 21. The static part is provided with
20 an upper static seal 33 which is enclosed in a stator 35 and co-acts with a lower rotatable seal 34. The insert holder 16 is mounted with bearings 22 on housing 18, 19, the liquid holder 7 is fastened to insert holder 16 by means of a mounting flange 23 and bolts 24. In foot 14 is formed an
25 outlet opening 26 which is closed by a valve 29 enclosed under pre-pressure of a pressure spring 27 and provided on its upper side with a locking nut 28. Foot 14 is provided with a number of seals 30 and a carrier ring 31 of a suitable material, for example rubber. In a filling apparatus
30 according to the invention the filler head 13 is displaceable in vertical direction above a horizontally disposed rotatable disc or turntable (not shown) in a manner such that the rotation axis 9 of liquid holder 7 and that of the turntable coincide. For filling purposes a semi-manufactured product 1
35 is laid on the turntable, wherein care is taken that the filling opening 6 in semi-manufactured product 1 extends round the rotating centre of the turntable. Filler head 13 is

then lowered onto semi-manufactured product 1, wherein valve 29 and closing spring 27 are pressed in, and liquid which is supplied via liquid inlet 21 and shaft hole 17, after setting into rotation the turntable, semi-manufactured product 1 and 5 rotatable parts of the filler head, is admitted from liquid holder 7 via outlet opening 26 and filling opening 6 into the space 5 in semi-manufactured product 1 where the liquid spreads in the manner set forth above in the description of figure 1. The rotatable parts of the filler head are set into 10 rotation by the semi-manufactured product 1 rotating with the turntable by means of the rubber carrier ring 31 in filler head 14. A stator pin 36 locks the stator 35 and the upper seal 33 against rotation. In order to prevent undesirable leakage of liquid to the bearings 22, a rotatable peripheral 15 edge 37 arranged on the upper part of insert 15 and a co-acting static peripheral edge 38 of complementary form connected to housing 19 form a labyrinth, and drains 39 are arranged in the upper cap 20 and housing 19.

It will be noted that this method provides the option of 20 simultaneous filling of a plurality of semi-manufactured products if the periphery of these semi-manufactured products is a regular polygon, and the filling openings of these semi-manufactured products are arranged such that they can all be placed in the centre of the turntable.

CLAIMS

1. Method for filling with liquid a semi-manufactured product (1) for a liquid-containing photovoltaic element, which semi-manufactured product (1) comprises at least one plate-like work electrode (2) and a plate-like counter-electrode (4) adhered thereto by means of a vapour and liquid-tight peripheral edge (3), wherein between the work electrode (2), the counter-electrode (4) and the peripheral edge (3) a space (5) is provided for receiving a liquid, comprising the steps of
- 10 (i) providing said semi-manufactured product (1),
(ii) arranging a filling opening (6) as desired in the work electrode (2) or the counter-electrode (4),
characterized by the steps of
- (iii) placing the semi-manufactured product (1) with the filling opening (6) directed upward on a horizontally placed rotatable disc in a manner such that the filling opening (6) extends round the rotation axis of the disc within a circle of a predetermined radius R,
- 15 (iv) placing around the filling opening (6) on the semi-manufactured product a holder (7) with a flat underside (14) and provided with an opening (26), wherein the opening (26) has at least an internal diameter $2R$ and the outer diameter of the holder (7) on its underside (14) is smaller than the smallest surface area dimension of the semi-manufactured
- 20 product (1),
- (v) introducing liquid into the holder (7) and causing rotation of the disc with the semi-manufactured product (1) and the holder (7) placed thereon at a rotation speed and for a period such that the space (5) in the semi-manufactured
- 30 product (1) is completely filled with liquid under the influence of centrifugal forces and the gravitational force acting on the liquid, and
- (vi) sealing the filling opening (6).
2. Method as claimed in claim 1, characterized in that

the holder (7) for placing in step (iv) is placed under pre-pressure on the semi-manufactured product.

3. Method as claimed in claims 1-2, **characterized in that** during placing of the holder (7) in step (iv) a liquid-tight
5 seal (30) is provided between the underside (14) of the holder (7) and the semi-manufactured product.

4. Method as claimed in any of the claims 1-3, **characterized in that** the rotation speed in step (v) amounts to at least 2000 rpm (revolutions per minute).

10 5. Method as claimed in claim 4, **characterized in that** the rotation speed in step (v) amounts to at least 4000 rpm (revolutions per minute).

6. Method as claimed in any of the claims 1-5, **characterized in that** a pulsating underpressure is applied in
15 the holder (7) during introducing of the liquid into the holder (7) in step (v).

7. Method as claimed in claim 6, **characterized in that** the underpressure amounts to about 10 kPa.

8. Method as claimed in any of the claims 1-7, wherein
20 the semi-manufactured product (1) comprises at least one glass substrate.

9. Apparatus for performing a method as claimed in claim 1, comprising a rotatable disc which in the operating situation is placed horizontally for placing thereon of a
25 semi-manufactured product (1) with a filling opening (6) directed upward, and a rotatable liquid holder (7) which can be placed around the filling opening (6) on the semi-manufactured product (1) and which is flat on its underside (14) and provided with an opening (26), wherein the outer
30 diameter of the holder (7) on its underside (14) is smaller than the smallest surface dimension of the semi-manufactured product.

10. Apparatus as claimed in claim 9, **characterized in that** the holder (7) is symmetrical on an axis of symmetry and
35 can be placed on a semi-manufactured product (1) in a manner such that the axis of symmetry coincides with the axis of rotation of the disc.

11. Apparatus as claimed in claim 10, characterized in that the holder is rotation-symmetrical.

12. Apparatus as claimed in any of the claims 9-11, characterized in that the interior of the holder (7) has in a
5 longitudinal section a downward widening conical progression.

13. Apparatus as claimed in any of the claims 9-12, characterized in that the holder (7) is provided with spring means (27) for placing the holder (7) under pre-pressure on the semi-manufactured product.

10 14. Apparatus as claimed in any of the claims 9-13, characterized in that the holder (7) is provided on its underside (14) with sealing means (30) for providing a liquid-tight seal between the underside of the holder (14) and the semi-manufactured product (1).

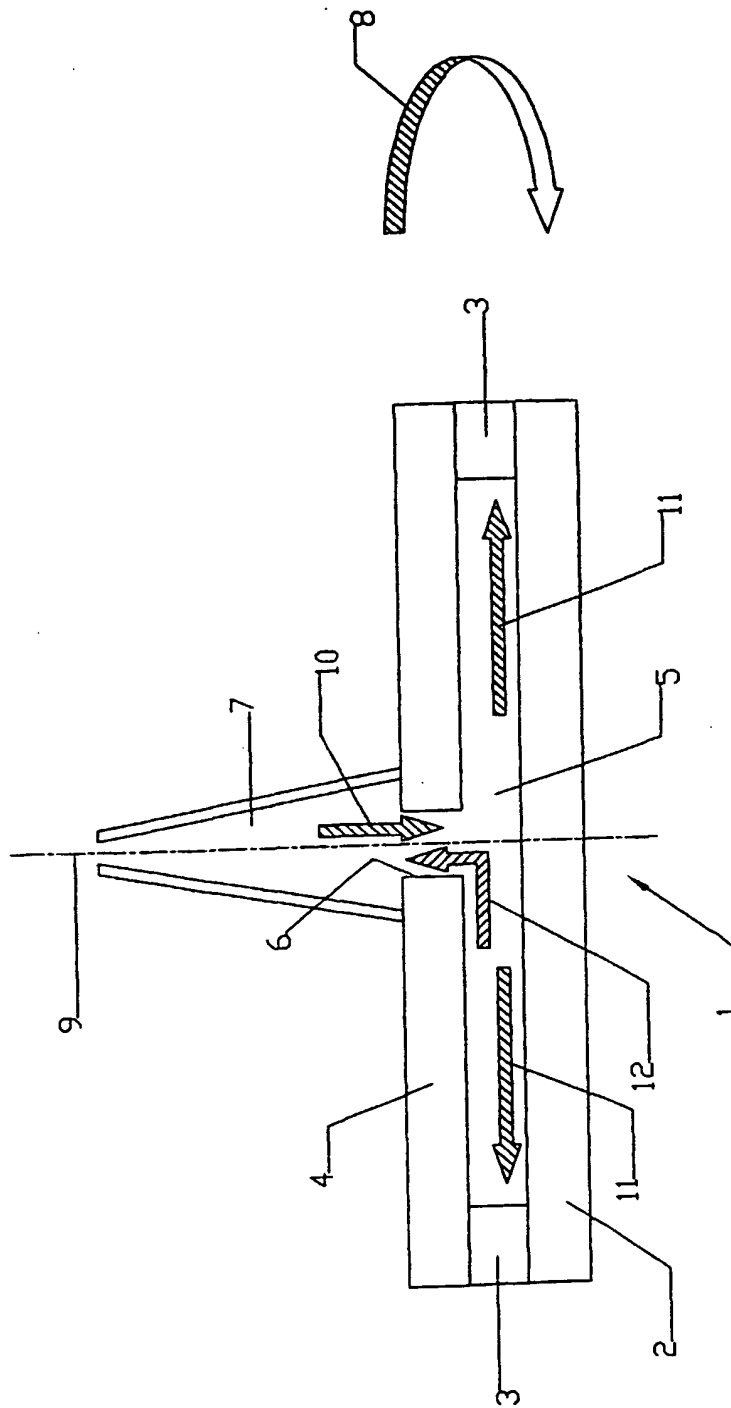


Fig. 1

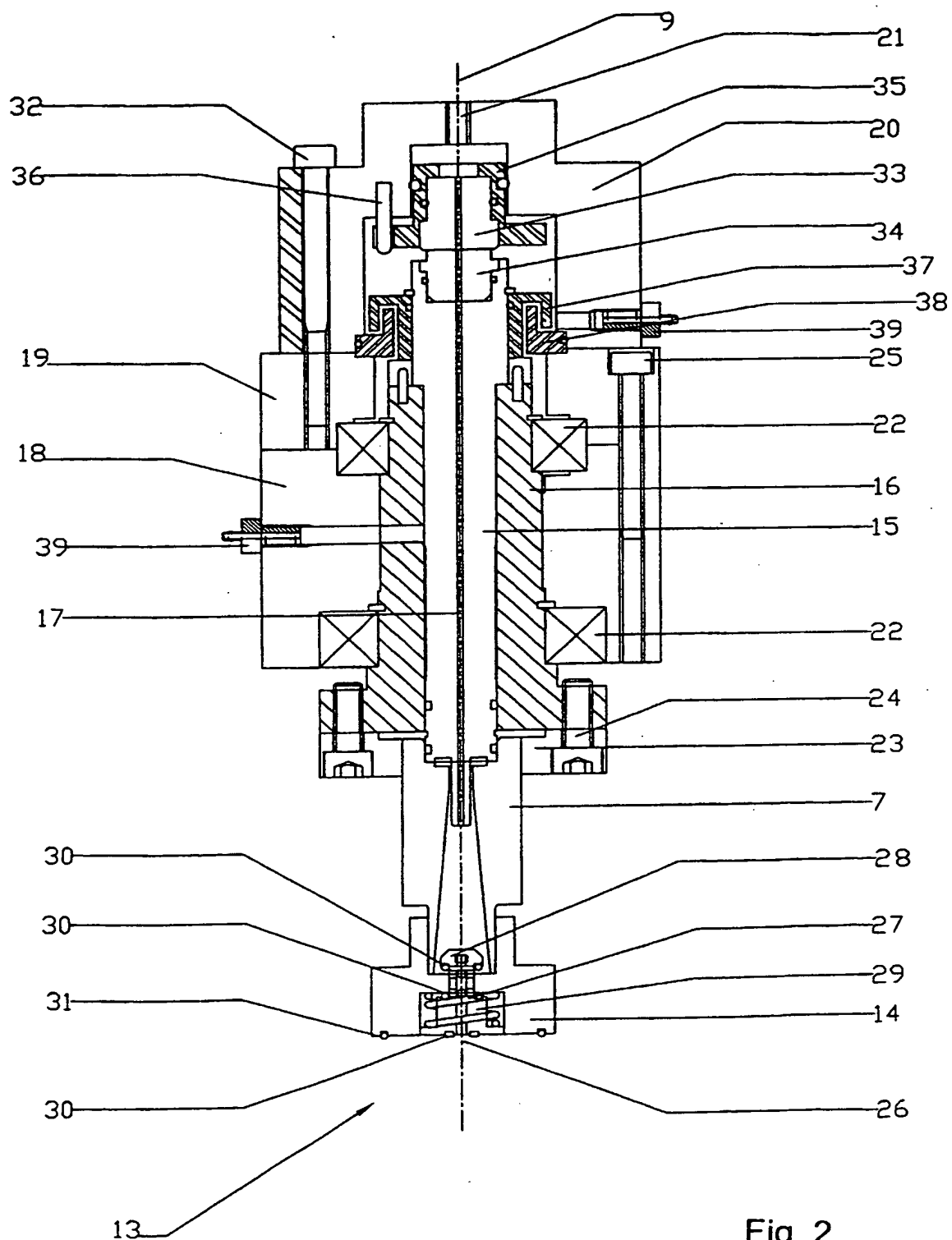


Fig. 2

INTERNATIONAL SEARCH REPORT

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A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 H01G9/20

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 H01G

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, INSPEC, PAJ, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 2001/004901 A1 (HAN LIYUAN ET AL) 28 June 2001 (2001-06-28) paragraph '0109! - paragraph '0110! ----	1
A	WO 00 62315 A (SKRYABIN IGOR LVOVICH ;PHANI GEORGE (AU); VITTORIO DAVID (AU); HOP) 19 October 2000 (2000-10-19) figures 2,3 ----	
A	WO 00 48212 A (KURTH GLAS & SPIEGEL AG ;KURTH MARTIN (CH)) 17 August 2000 (2000-08-17) page 7, line 26 -page 8, line 11 -----	

☐ Further documents are listed in the continuation of box C.☒ Patent family members are listed in annex.

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- *X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
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Patent document cited in search report		Publication date	Patent family member(s)		Publication date
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			AU	761366 B2	05-06-2003
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			EP	1192627 A1	03-04-2002
			JP	2002541641 T	03-12-2002
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			AU	2274600 A	29-08-2000
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			DE	50001095 D1	20-02-2003
			EP	1155424 A1	21-11-2001
			JP	2002536805 T	29-10-2002
			US	6462266 B1	08-10-2002
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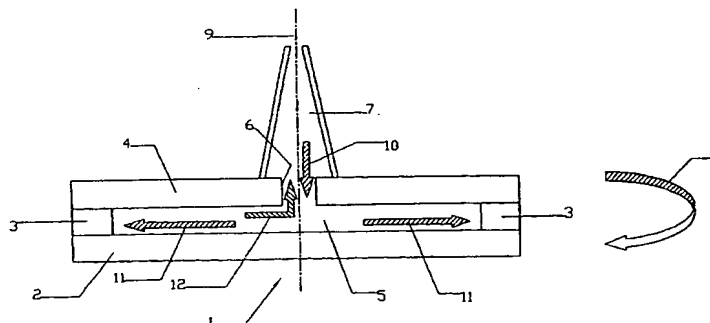
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CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH,
GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC,
LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW,
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(54) Title: METHOD AND APPARATUS FOR FILLING A DYE SENSITIZED SOLAR CELL WITH ELECTROLYTE



(57) Abstract: Method for filling with liquid a semi-manufactured product for a liquid-containing photovoltaic element, which semi-manufactured product comprises at least one plate-like work electrode and a plate-like counter-electrode adhered thereto by means of a vapour and liquid-tight peripheral edge, wherein between the work electrode, the counter-electrode and the peripheral edge a space is provided for receiving a liquid, comprising the steps of (i) providing said semi-manufactured product, (ii) arranging a filling opening as desired in the work electrode or the counter-electrode, (iii) placing the semi-manufactured product with the filling opening directed upward on a horizontally placed rotatable disc in a manner such that the filling opening extends round the rotation axis of the disc within a circle of a predetermined radius R, (iv) placing around the filling opening on the semi-manufactured product a holder with a flat underside and provided with an opening, wherein the opening has at least an internal diameter 2R and the outer diameter of the holder on its underside is smaller than the smallest surface area dimension of the semi-manufactured product, (v) introducing liquid into the holder and causing rotation of the disc with the semi-manufactured product and the holder placed thereon at a rotation speed and for a period such that the space in the semi-manufactured product is completely filled with liquid under the influence of centrifugal forces and the gravitational force acting on the liquid, and (vi) sealing the filling opening, and apparatus for performing this method.

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(15) Information about Correction:
see PCT Gazette No. 24/2004 of 10 June 2004, Section II

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